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First Named Inventor: Waldrop, Stephanie D.  
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Group Art Unit: 1714  
Examiner Name: Anthony, Joseph D.

**SUPPLEMENTAL DECLARATION FOR UTILITY PATENT APPLICATION  
UNDER 37 CFR 1.67**

As below named inventors, we hereby declare that:

Our residences, mailing addresses and citizenships are as stated below next to our names. We believe that we, the Applicants, are the original, first and joint-inventors of the subject matter which is claimed and for which a patent is sought for the invention entitled "Nonflammable Ethylene Oxide Gas Blend Compositions and Method Of Making Same" (as amended), the specification of which was filed on 12/05/2003, application number 10/729,133. The subject matter below was part of our invention, proving the synergy disclosed and claimed therein, and was invented before the filing date of the original application, as above identified, for such invention.

We hereby request that the examiner take notice of the attachments hereto, which are described in our response to the Non-final Office Action of November 17, 2004:

The following descriptive text and attached graphs and tables accurately describe and depict ternary flammability data for compositions of ethylene oxide, carbon dioxide and trifluoriodomethane in our invention, application 10/729,133, "Nonflammable Ethylene Oxide Gas Blend Compositions and Method Of Making Same" (as amended), filed December 5, 2003.

Jonathan S. Nimitz, undersigned is also the first named inventor of Nimitz et al. U.S. Patent No. 5,674,451, and thus is familiar with the information disclosed therein. The data provided here, that resulted in the filing of U.S. Patent Appl. No. 10/729,133, were obtained subsequent to issuance of Nimitz et al., U.S. Patent No. 5,674,451, and were obtained after extensive experimentation, over several years, costing hundreds of thousands of dollars. Had such synergistic results been obvious, there would have been no need to spend the extensive time and effort in finding that the compositions disclosed in U.S. Patent Appl. No. 10/729,133 were synergistic.

**Description of Data Revealing Disclosed and Claimed Synergistic Invention of  
Ethylene Oxide by a Combination of Carbon Dioxide with CF<sub>3</sub>I.**

Ethylene oxide (EO) is a highly explosive gas, even in the absence of air or oxygen. Its flammability limits (in air) are 3% (the lower flammability limit) to

100% (the upper flammability limit, i.e. pure EO). Addition of a sufficient ratio of inerting agents to EO can eliminate this flammability. For example, historically, 88% by weight dichlorodifluoromethane (Freon 12) was used to inert 12% EO to create a nonflammable sterilant gas blend. By creating this 88/12 mixture, flammability was eliminated at all concentrations in air (e.g., all dilutions of the 88/12 blend). The 88/12 blend was phased out of production because of the stratospheric ozone depletion caused.

We have investigated the uses of carbon dioxide,  $\text{CF}_3\text{I}$ , and blends of these two flammability suppressants for inerting EO. We have separately tested the concentrations of carbon dioxide and  $\text{CF}_3\text{I}$  required to inert EO in our laboratory using the apparatus and procedures specified in American Society for Testing and Materials (ASTM) test number E-681 ("Standard Test Method for Concentration Limits of Flammability of Chemicals"). We have found that in order to inert EO with carbon dioxide (i.e., to create a blend that is nonflammable at all dilutions in air), a ratio of at least 91.5% carbon dioxide (with a maximum of 8.5% EO) is needed. Note: this and all subsequent ratios are mole ratios. We have also found that in order to inert EO with  $\text{CF}_3\text{I}$  at all dilutions in air, a ratio of at least 96%  $\text{CF}_3\text{I}$  (with a maximum of 4% EO) is needed. These data indicate that individually both carbon dioxide and  $\text{CF}_3\text{I}$  are relatively poor inerting agents for EO.

A ternary flammability diagram shows regions of flammable and nonflammable mixtures for both two- and three-component blends. The ratios of two components needed for inertion are shown as intercepts along the axes. Figure 1 shows these intercepts occurring at ratios of 91.5/8.5 for binary  $\text{CO}_2/\text{EO}$  blends and at 96/4 for binary  $\text{CF}_3\text{I}/\text{EO}$  blends. We have also tested numerous ternary blends of EO, carbon dioxide, and  $\text{CF}_3\text{I}$  to determine the flammability of these ternary blends and the ratios at which flammability is inerted at all concentrations in air.

To obtain these data, the selected ratios of the components were blended then tested for flammability by ASTM E-681 at several dilutions in air. A typical data sheet for a series of tests of a particular blend (95/5 blend of  $\text{CO}_2/\text{CF}_3\text{I}$  with 24% EO at several dilutions in air) is shown in Figure 2. The data from a group of tests using the 95/5 blend with varying concentrations of EO are summarized in Figure 3. The set of data from Figure 3 are plotted in Figure 4. In Figure 4, the slope of the line from the origin asymptotic to the flammable region gives the ratio for the borderline of nonflammability. The result of the series of tests shown in Figures 2 and 3 is one point on the ternary flammability diagram in Figure 1.

In the cases of ternary blends where no synergism (i.e., more effective inertion than linearly predicted) occurs, the borderline between the flammable and nonflammable regions on the ternary flammability diagram is a straight line between the two intercepts obtained from the binary mixtures. The summary of our testing of hundreds of ternary blends of EO with  $\text{CO}_2$  and  $\text{CF}_3\text{I}$  is shown in Figure 1. This figure reveals very large curvature to the flammability borderline.

This curvature indicates much more effective flammability inertion of EO by blends of carbon dioxide with CF<sub>3</sub>I than would have been predicted linearly. In other words, strong synergism occurs when a small amount of CF<sub>3</sub>I is added to carbon dioxide. This result was never previously reported, could not have been predicted, and has led to a new and valuable invention. It shows that by blending EO with carbon dioxide plus small amounts of CF<sub>3</sub>I, nonflammable sterilant blends containing large proportions of EO can be produced inexpensively and without contributing to stratospheric ozone depletion or global warming.

We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 3-14-05

Jonathan S. Nimitz

Inventor: Jonathan S. Nimitz  
Residence Address: 3300 Mountain Rd. NE, Albuquerque, NM 87106  
Mailing Address: Same  
Citizenship USA

Stephanie D. Waldrop

Inventor: Stephanie D. Waldrop  
Residence Address: 8109 Rancho Seguro NW, Albuquerque, NM 87120  
Mailing Address: Same  
Citizenship USA

Edward T. McCullough

Inventor: Edward T. McCullough  
Residence Address: 4913 Hilton Ave. NE, Albuquerque, NM 87110  
Mailing Address: Same  
Citizenship USA

Patrick M. Ohooge

Inventor: Patrick M. Ohooge  
Residence Address: 592 Dixon Rd., Corrales, NM 87048  
Mailing Address: P.O. Box 1731, Corrales, NM 87048  
Citizenship USA

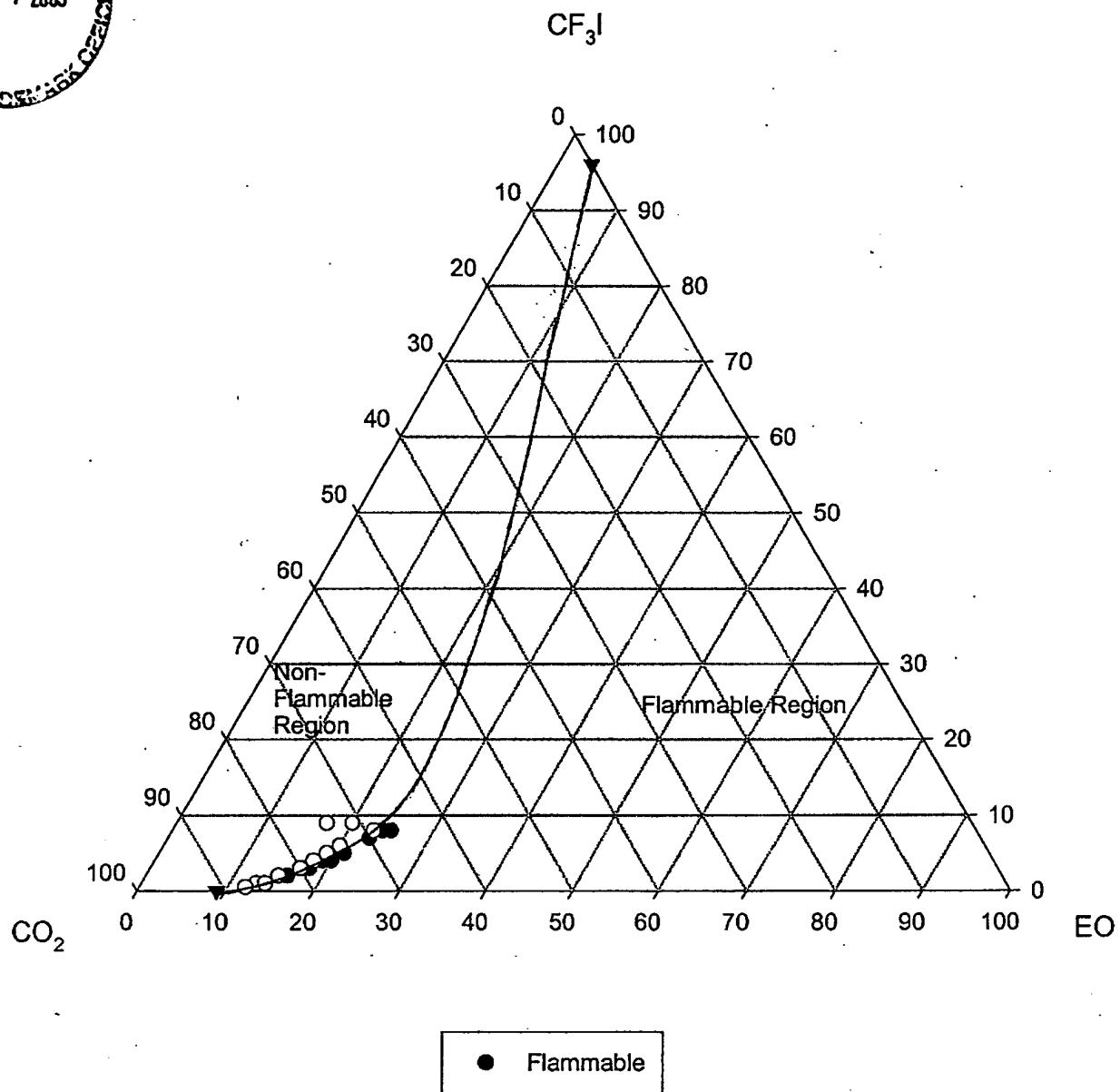
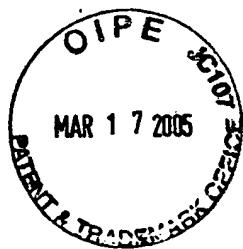


Figure 1. Ternary flammability diagram for blends of CO<sub>2</sub>, CF<sub>3</sub>I, and EO. Outline circles are blends that tested nonflammable. Filled circles are blends that tested flammable.



Figure 2.

FLAMMABILITY DATA  
 24% Ethylene Oxide 3.8% CF3I 72.2% CO<sub>2</sub>  
 (Molar Ratio of CO<sub>2</sub> to CF3I 95/5)

Actual pressure reading (torr)	667	667	667	667	667	667
Zeroed pressure reading (torr)	33	33	33	33	33	33
Atmospheric pressure (torr)	634	634	634	634	634	634
Desired total concentration of blend (%)	15	20	25	30	30	35
Desired torr of blend to add	95	127	159	190	190	222
Drift rate (torr in 60 sec)	0	0	0	0	0	0
Actual start torr	37	36	38	36	36	37
Desired final torr (start + desired + drift)	132	163	197	226	226	259
Actual final torr	132	163	196	225	225	259
Actual torr added (final - start - drift)	95	127	158	189	189	222
Water added (microliters)	92	87	81	76	76	71
Less needle volume	54	54	54	54	54	54
Syringe reading for water	38	33	27	22	22	17
Actual concentration of blend	15.0%	20.0%	24.9%	29.8%	29.8%	35.0%
Observations	1" flame	3" flame	full flask	1/4 flask	3" flame	
Results (positive or negative)	negative	negative	positive	positive	positive	negative



Figure 3.  
FLAMMABILITY DATA  
Ethylene Oxide, CF<sub>3</sub>I and CO<sub>2</sub> (5:95)

Initial Mixture			% Mixture in Air	In Air				
%CF <sub>3</sub> I	%CO <sub>2</sub>	%EtO		%CF <sub>3</sub> I	%CO <sub>2</sub>	Total Non-Flammable	%EtO	Flammable?
3.8%	72.2%	24.0%	24.9%	0.9%	18.0%	18.9%	6.0%	Y
3.8%	72.2%	24.0%	29.8%	1.1%	21.5%	22.6%	7.2%	Y
3.9%	74.1%	22.0%	26.8%	1.0%	19.9%	20.9%	5.9%	Y
3.8%	72.2%	24.0%	15.0%	0.6%	10.8%	11.4%	3.6%	N
3.8%	72.2%	24.0%	20.0%	0.8%	14.4%	15.2%	4.8%	N
3.8%	72.2%	24.0%	35.0%	1.3%	25.3%	26.6%	8.4%	N
3.9%	74.1%	22.0%	21.6%	0.8%	16.0%	16.8%	4.8%	N
3.9%	74.1%	22.0%	21.8%	0.9%	16.2%	17.0%	4.8%	N
3.9%	74.1%	22.0%	24.0%	0.9%	17.8%	18.7%	5.3%	N
3.9%	74.1%	22.0%	32.0%	1.2%	23.7%	25.0%	7.0%	N
4.0%	76.0%	20.0%	15.1%	0.6%	11.5%	12.1%	3.0%	N
4.0%	76.0%	20.0%	19.9%	0.8%	15.1%	15.9%	4.0%	N
4.0%	76.0%	20.0%	24.8%	1.0%	18.8%	19.8%	5.0%	N
4.0%	76.0%	20.0%	27.0%	1.1%	20.5%	21.6%	5.4%	N
4.0%	76.0%	20.0%	28.1%	1.1%	21.4%	22.5%	5.6%	N
4.0%	76.0%	20.0%	30.0%	1.2%	22.8%	24.0%	6.0%	N
4.0%	76.0%	20.0%	34.9%	1.4%	26.5%	27.9%	7.0%	N



Figure 4.  
Inertion Diagram  
Ethylene Oxide, CF<sub>3</sub>I and CO<sub>2</sub> (5:95)

